

September 1998

Technology Demonstration Plan

Evaluation of Polychlorinated Biphenyl (PCB) Field Analytical Techniques

EnviroLogix, Inc.

Sponsored by:

U. S. Environmental Protection Agency National Exposure Research Laboratory Las Vegas, NV 89193-3478

Prepared by:

Oak Ridge National Laboratory Lockheed Martin Energy Research Corporation Oak Ridge, TN 37831-6120



APPROVAL SIGNATURES

This document is intended to ensure that all aspects of the demonstration are documented, scientifically sound, and that operational procedures are conducted within quality assurance/quality control specifications and health and safety regulations.

The signatures of the individuals below indicate concurrence with, and agreement to operate compliance with, procedures specified in this document.

U. S. ENVIRONMENTAL PROTECTION AGENCY

Project Manager:		
	Eric Koglin	Date
Technical Lead:		
	Deana Crumbling	Date
External Reviewer:		
	Barry Lesnick	Date
	OAK RIDGE NATIONAL LABORATO	ORY
Program Manager: _		
	Roger Jenkins, Ph.D.	Date
Technical Lead:		
	Amy Dindal	Date
QA Specialist:		
	Janet Wagner	Date
Statistician:		
	Charles Bayne, Ph. D.	Date
U. S. D	EPARTMENT OF ENERGY, Oak Ridge Op	perations Office
Program Manager:		
	Regina Chung	Date

Technical Lead:		
	David Carden	Date
	SITE APPROVAL	
ES&H Coordinator: _	Fred Smith	Date
	TECHNOLOGY DEVELOPER	
EnviroLogix, Inc.:	Jonathan Matt	

FORWARD

The U.S. Environmental Protection Agency (EPA) is charged by Congress with protecting the nation's natural resources. The National Exposure Research Laboratory (NERL) is EPA's center for the investigation of technical and management approaches for identifying and quantifying risks to human health and the environment. NERL's research goals are to (1) develop and evaluate technologies for the characterization and monitoring of air, soil, and water; (2) support regulatory and policy decisions; and (3) provide the science support needed to ensure effective implementation of environmental regulations and strategies.

EPA created the Environmental Technology Verification (ETV) Program to facilitate the deployment of innovative technologies through performance verification and information dissemination. The goal of the ETV Program is to further environmental protection by substantially accelerating the acceptance and use of improved and cost-effective technologies. The ETV Program is intended to assist and inform those involved in the design, distribution, permitting, and purchase of environmental technologies. This program is administered by NERL's Environmental Sciences Division in Las Vegas, Nevada.

The U.S. Department of Energy's (DOE) Environmental Management (EM) program has entered into active partnership with EPA, providing cooperative technical management and funding support. DOE EM realizes that its goals for rapid and cost effective cleanup hinges on the deployment of innovative environmental characterization and monitoring technologies. To this end, DOE EM shares the goals and objectives of the ETV.

Candidate technologies for these programs originate from the private sector and must be commercially ready. Through the ETV Program, developers are given the opportunity to conduct rigorous demonstrations of their technologies under realistic field conditions. By completing the evaluation and distributing the results, EPA establishes a baseline for acceptance and use of these technologies.

TABLE OF CONTENTS

FORW	/ARD .	iii
EXEC	UTIVE	SUMMARY vii
ABBR	EVIATI	ONS AND ACRONYMS viii
1.0	INTRO	DDUCTION
	1.1	Demonstration Objectives
	1.2	What is the Environmental Technology Verification (ETV) Program?
	1.3	Technology Verification Process
		1.3.1 Needs Identification and Technology Selection
		1.3.2 Demonstration Planning and Implementation
		1.3.3 Report Preparation
		1.3.4 Information Distribution
	1.4	Purpose of this Demonstration Plan
2.0	DEMO	ONSTRATION RESPONSIBILITIES AND COMMUNICATION
	2.1	Demonstration Organization and Participants
	2.2	Organization
	2.3	Responsibilities
3.0	TECH	NOLOGY DESCRIPTION
2.0	3.1	EnviroLogix PCB in Soil Tube Kit
	0.1	3.1.1 Principle
		3.1.2 Applications and Advantages
		3.1.3 Procedure
		3.1.3.1 Materials
		3.1.3.2 Extraction
		3.1.3.3 Assay
		3.1.3.4 Interpreting Results
		3.1.3.5 Precautions and Notes
		3.1.4 Sensitivity and Cross-Reactivity
	3.2	Demonstration Performance Goals
4.0	DEMC	ONSTRATION SITE AND SAMPLE DESCRIPTIONS
4.0	4.1	Site Name and Location
	4.2	Site History
	4.3	Site Characteristics
	4.4	Soil Sample Descriptions
	-⊤ T	4.4.1 Oak Ridge Reservation, Portsmouth, and Paducah Soils
		4.4.2 Tennessee Reference Soil
	4.5	Extract Sample Descriptions
	4.6	Sample Stability Study
	7.0	Dumple Ducomity Diddy

5.0	CON	FIRMATORY PROCESS	14
	5.1	Method Selection	14
	5.2	Reference Laboratory Selection	14
	5.3	In-Field Support Laboratory	
	5.4	Special QC Requirements	
	5.5	Laboratory Audit	
		·	
6.0	PCB	SOIL SAMPLE COLLECTION	
	6.1	Sample Collection Plan	15
		6.1.1 Sample Collection Procedures	15
	6.2	Preliminary Soil Characterization	
	6.3	Predemonstration Sample Preparation, Distribution, and Analysis	16
		6.3.1 Predemonstration Sample Distribution	17
		6.3.2 Predemonstration Sample Analysis Results	17
	6.4	Sample Preparation for Demonstration	17
	6.5	Sample Labeling for Demonstration	17
7.0		IONSTRATION DESIGN	
	7.1	Objectives	
	7.2	Experimental Performance Measures	
		7.2.1 Qualitative Performance measures	
		7.2.2 Quantitative Performance measures	
	7.3	Experimental Factors	
		7.3.1 Glossary of Terms	
		7.3.2 Summary of Demonstration Activities	
	7.4	Field Data	23
		7.4.1 Field Audit	23
	7.5	Demonstration Schedule	23
	7.6	Field Operations	23
		7.6.1 Communication and Documentation	23
		7.6.2 Sample Distribution	23
		7.6.2.1 Archive Samples	23
	7.7	Statistical Analysis of Results	24
0.0	OTTA	A VEW A COLUD A MORE DE O VECET DE A M	2.5
8.0	_	LITY ASSURANCE PROJECT PLAN	
	8.1	Purpose and Scope	
	8.2	Quality Assurance Responsibilities	
	8.3	Data Quality Indicators	
		8.3.1 Representativeness	
		8.3.2 Comparability	
		8.3.3 Completeness	
		8.3.4 Accuracy	
		8.3.5 Precision	
	8.4	Calibration Procedures and Quality Control Checks	
		8.4.1 Initial Calibration Procedures	
		8.4.2 Continuing Calibration Procedures	
		8.4.3 Method Blanks	27

		8.4.4 Spike Samples	27
		8.4.5 Laboratory Control Samples	
		8.4.6 Performance Evaluation Materials	
		8.4.7 Replicate Samples	28
	8.5	Data Reduction, Review, and Reporting	
		8.5.1 Data Reduction	
		8.5.2 Data Review	
		8.5.3 Data Reporting	29
	8.6	Calculation of Data Quality Indicators	
	8.7	Performance and System Audits	
		8.7.1 Performance Audit	
		8.7.2 On-Site System Audits	
	8.8	Quality Assurance Reports	
		8.8.1 Status Reports	
		8.8.2 Audit Reports	
	8.9	Corrective Actions	
9.0	DATA	MANAGEMENT AND ASSESSMENT	31
10.0	HEAL'	TH AND SAFETY PLAN	31
	10.1	Introduction	31
	10.2	Contact Information	
	10.3	Health and Safety Plan Enforcement	32
	10.4	Site Background	
	10.5	Visitors	
	10.6	Demonstration-Specific Hazard Evaluation	
	10.7	Training Requirements	
	10.8	Exposure Pathways	
	10.9	Health Effects	
	10.10	Physical Hazards	
	10.11	Personal Protection	
	10.12	Site Control	
REFEI	RENCES	S	36
Appen	dix A O	ak Ridge Sample Management Office Procedures	37
Appen	dix B R	eference Laboratory Analytical Procedures	6(
Appen	dix C In	n-Field Support Laboratory Analytical Procedures	96
Appen	dix D R	&D Soil Sample Collection Plan	18
Annen	dix E. Pr	re-demonstration Instructions to Developer	26

EXECUTIVE SUMMARY

EPA created the Environmental Technology Verification (ETV) Program to facilitate the deployment of innovative technologies through performance verification and information dissemination. The goal of the ETV Program is to further environmental protection by substantially accelerating the acceptance and use of improved and cost-effective technologies. The ETV Program is intended to assist and inform those involved in the design, distribution, permitting, and purchase of environmental technologies. This program is administered by the EPA's National Exposure Research Laboratory in Las Vegas, Nevada. This technology demonstration plan has been developed to describe the verification of EnviroLogix's PCB in Soil Tube Assay, which is a semi-quantitative immunoassay technique. The Oak Ridge National Laboratory (ORNL) will serve as the verification organization for the demonstration. ORNL's role is to provide technical and administrative leadership and support in conducting the demonstration.

The purpose of this demonstration is to obtain performance information regarding the test kit, to compare the results to conventional fixed-laboratory results, and to provide supplemental information (e.g., cost, sample throughput, and training requirements) regarding the operation of the technology. The demonstration will be conducted under two climatic conditions. One set of activities will be conducted outdoors, with naturally fluctuating temperatures and relative humidity conditions. A second set will be conducted in a controlled environmental facility, with lower, relatively stable temperatures and relative humidities. Multiple soil types, collected from sites in Ohio, Kentucky, and Tennessee, will be used in this study. PCB soil concentrations will range from approximately 0.1 to 700 parts per million (ppm). The developer will also analyze 24 solutions of known PCB concentration that will simulate extracted wipe samples. The extract samples will range in concentration from 0 to 100 μ g/mL.

ABBREVIATIONS AND ACRONYMS

CASD	Chemical and Analytical Sciences Division	
CSCT	Consortium for Site Characterization Technology	
DOE	U. S. Department of Energy	
ELISA	enzyme linked immunosorbent assay	
EPA	U. S. Environmental Protection Agency	
ERA	Environmental Resource Associates	
ESD-LV	Environmental Science Division-Las Vegas	
ESH&Q	Environmental Safety, Health, and Quality	
ETTP	East Tennessee Technology Park	
ETV	Environmental Technology Verification Program	
ETVR	Environmental Technology Verification Report	
fn	false negative result	
fp	false positive result	
GC	gas chromatography	
HASP	Health and Safety Plan	
LCS	Laboratory Control Sample	
LMER	Lockheed Martin Energy Research	
LMES	Lockheed Martin Energy Systems	
MS/MSD	matrix spike/matrix spike duplicate	
OD	optical density	
ORNL	Oak Ridge National Laboratory	
ORNL-GJ	Oak Ridge National Laboratory, Grand Junction, Colorado	
PARCC	precision, accuracy, representativeness, completeness, comparability	
PCBs	polychlorinated biphenyls	
PE	performance evaluation	
PPE	personal protective equipment	

ppm	parts per million, mg/kg for soils or µg/mL for extracts	
QA	quality assurance	
QAPP	Quality Assurance Project Plan	
QC	quality control	
RFD	request for disposal	
RSD	relative standard deviation	
SCM	Site Characterization and Monitoring Technology Pilot	
SITE	Superfund Innovative Technology Evaluation program	
SMO	Sample Management Office	
SOP	standard operating procedure	
SOW	statement of work	
SSM	synthetic soil matrix	
SVOCs	semivolatile organic compounds	
VOCs	volatile organic compounds	

1.0 INTRODUCTION

This chapter discusses the purpose of the demonstration and the demonstration plan, describes the elements of the demonstration plan, and provides an overview of the Environmental Technology Verification (ETV) Program and the technology verification process.

1.1 Demonstration Objectives

The purpose of this demonstration is to evaluate the performance of the EnviroLogix PCB in Soil Tube Kit. The kit is a semi-quantitative immunoassay technology that evaluates polychlorinated biphenyls (PCBs) in the field. The Oak Ridge National Laboratory (ORNL) will serve as the verification organization for the demonstration. Specifically, this plan defines the following elements of the demonstration:

- Roles and responsibilities of demonstration participants;
- Procedures governing demonstration activities such as sample collection, preparation, analysis, data collection, and interpretation;
- Experimental design of the demonstration;
- Quality assurance (QA) and quality control (QC) procedures for conducting the demonstration and for assessing the quality of the data generated from the demonstration; and,
- Health and safety requirements for performing work at hazardous waste sites.

1.2 What is the Environmental Technology Verification (ETV) Program?

The Environmental Technology Verification (ETV) Program was created by the Agency to facilitate the deployment of innovative technologies through performance verification and information dissemination. The goal of the ETV Program is to further environmental protection by substantially accelerating the acceptance and use of improved and cost-effective technologies. The ETV Program is intended to assist and inform those involved in the design, distribution, permitting, and purchase of environmental technologies. The ETV Program capitalizes upon and applies the lessons that were learned in the implementation of the Superfund Innovative Technology Evaluation (SITE) Program to the verification of twelve categories of environmental technology: Drinking Water Systems, Pollution Prevention/Waste Treatment, Pollution Prevention/ Innovative Coatings and Coatings Equipment, Indoor Air Products, Air Pollution Control, Advanced Monitoring Systems, EvTEC (an independent, private-sector approach), Wet Weather Flow Technologies, Pollution Prevention/Metal Finishing, Source Water Protection Technologies, Site Characterization and Monitoring (SCM) Technology (also referred to as Consortium for Site Characterization Technology (CSCT)), and Climate Change Technologies. This demonstration will be administered by SCM/CSCT.

1.3 Technology Verification Process

The technology verification process is intended to serve as a template for conducting technology demonstrations that will generate high-quality data which EPA can use to verify technology performance. Four key steps are inherent in the process:

Needs identification and technology selection

- Demonstration planning and implementation
- Report preparation
- Information distribution

1.3.1 Needs Identification and Technology Selection

The first aspect of the technology verification process is to determine technology needs of the EPA and the regulated community. EPA, the U.S. Department of Energy, the U.S. Department of Defense, industry, and state agencies are asked to identify technology needs and interest in a technology. Once a technology need is established, a search is conducted to identify suitable technologies that will address this need. The technology search and identification process consists of reviewing responses to *Commerce Business Daily* announcements, searches of industry and trade publications, attendance at related conferences, and leads from technology developers. Characterization and monitoring technologies are evaluated against the following criteria:

- Meets user needs
- May be used in the field or in a mobile laboratory
- Applicable to a variety of environmentally impacted sites
- High potential for resolving problems for which current methods are unsatisfactory
- Costs are competitive with current methods
- Performance is better than current methods in areas such as data quality, sample preparation, or analytical turnaround time
- Uses techniques that are easier and safer than current methods
- Is a commercially available, field-ready technology

1.3.2 Demonstration Planning and Implementation

After a technology has been selected, EPA, the verification organization, and the developer agree to the responsibilities for conducting the demonstration and evaluating the technology. The following issues are addressed at this time:

- Identifying demonstration sites that will provide the appropriate physical or chemical environment, including contaminated media
- Identifying and defining the roles of demonstration participants, observers, and reviewers
- Determining logistical and support requirements (for example, field equipment, power and water sources, mobile laboratory, communications network)
- Arranging analytical and sampling support

 Preparing and implementing a demonstration plan that addresses the experimental design, sampling design, quality assurance/quality control (QA/QC), health and safety considerations, scheduling of field and laboratory operations, data analysis procedures, and reporting requirements

1.3.3 Report Preparation

Innovative technologies are evaluated independently and, when possible, against conventional technologies. The field technologies are operated by the developers in the presence of independent technology observers. The technology observers are provided by EPA or a third party group. Demonstration data are used to evaluate the capabilities, limitations, and field applications of each technology. Following the demonstration, all raw and reduced data used to evaluate each technology are compiled into a technology evaluation report, which is mandated by EPA as a record of the demonstration. A data summary and detailed evaluation of each technology are published in an ETVR.

1.3.4 Information Distribution

The goal of the information distribution strategy is to ensure that ETVRs are readily available to interested parties through traditional data distribution pathways, such as printed documents. Documents are also available on the World Wide Web through the ETV Web site (http://www.epa.gov/etv) and through a Web site supported by the EPA Office of Solid Waste and Emergency Response's Technology Innovation Office (http://CLU-in.com).

1.4 Purpose of this Demonstration Plan

The purpose of the demonstration plan is to describe the procedures that will be used to verify the performance goals of a technology. This document incorporates the QA/QC elements needed to provide data of appropriate quality sufficient to reach a defensible position regarding the technology performance. This is not a method validation study, nor does it represent every environmental situation which may be acceptable for this technology. But it will provide data of sufficient quality to make a judgement about the application of the technology under conditions similar to those encountered in the field demonstration.

2.0 DEMONSTRATION RESPONSIBILITIES AND COMMUNICATION

This section identifies the organizations involved in this demonstration and describes the primary responsibilities of each organization. It also describes the methods and frequency of communication that will be used in coordinating the demonstration activities.

2.1 Demonstration Organization and Participants

Participants in this demonstration are listed in Table 2-1. The specific responsibilities of each demonstration participant are discussed in Section 2.3 This demonstration is being coordinated by the Oak Ridge National Laboratory (ORNL) under the direction of the U.S. Environmental Protection Agency's (EPA) Office of Research and Development, National Exposure Research Laboratory, Environmental Sciences Division - Las Vegas, Nevada (ESD-LV) and the U.S. Department of Energy's Environmental Management Program, Oak Ridge Operations Office, Oak Ridge, Tennessee. ESD-LV and DOE's roles are to administer the demonstration program. ORNL's role is to provide technical and administrative leadership and support in conducting the demonstration. EnviroLogix Inc. is the technology developer participating in this demonstration.

Table 2-1. Demonstration Participants PCB Field Analytical Technology Demonstration

Organization	Point(s) of Contact	Role
Oak Ridge National Laboratory P.O. Box 2008 Bethel Valley Road Bldg. 4500S, MS-6120 Oak Ridge, TN 37831-6120	Program Manager: Roger Jenkins phone: (423) 576-8594 fax: (423) 576-7956 email: jenkinsra@ornl.gov Technical Lead: Amy Dindal phone: (423) 574-4863 fax: (423) 576-7956 email: dindalab@ornl.gov Site Operations/ESH&Q: Fred Smith phone: (423) 574-4945 fax: (423) 574-6721 email: smithfj@ornl.gov	verification organization
U. S. EPA National Exposure Research Laboratory Environmental Science Division P.O. Box 93478 Las Vegas, NV 89193-3478 Technology Innovation Office 401 M Street, SW (5102G) Washington, DC 20460	Project Officer: Eric Koglin phone: (702) 798-2432 fax: (702) 798-2261 email: Koglin-Eric@wpmail.las.epa.gov Technical Lead: Deana Crumbling phone: (703) 603-0643 fax: (703) 603-9135 email: crumbling.deana@epamail.epa.gov	EPA project management
U. S. DOE ORNL Site Office P.O. Box 2008 Bldg. 4500N, MS-6269 Oak Ridge, TN 37831-6269 Oak Ridge Operations Office Three Main Street Oak Ridge, TN 37830	Program Coordinator: Regina Chung phone: (423) 576-9902 fax: (423) 574-9275 email: chungr@ornl.gov Technical Program Manager: David Carden phone: (423) 576-9262 fax: (423) 576-6074 email: cardendm@oro.doe.gov	DOE/ORO project management team
EnviroLogix, Inc. 55 Industrial Way Portland, ME 04103	Jonathan Matt phone: (207) 797-0300 fax: (207) 797-7533 email: elix4@aol.com	technology developer
Oak Ridge National Laboratory 2597 B-3/4 Road Grand Junction, CO 81503	Frank Gardner phone: (970) 248-6238 fax: (970) 248-6147 email: fgg@ornl.gov	in-field support laboratory
LAS Laboratories ^a 975 Kelly Johnson Drive Las Vegas, NV 89119 ^a No longer in business	Mary Ford phone: (702) 361-3955 fax: (702) 361-8146 email: m_ford@LASNVLAB.COM	reference laboratory

2.2 Organization

In Figure 2-1 is presented an organizational chart depicting the lines of communication for the demonstration. Note that the double-arrow lines signify that each participant is encouraged to openly communicate with other members of the demonstration team.

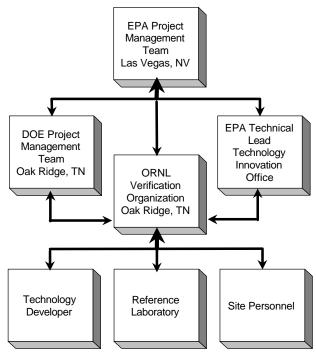


Figure 2-1. Organization Chart. Refer to Table 2-1 for specific names of individuals performing each task.

2.3 Responsibilities

The following is a delineation of each participant's responsibilities for the demonstration. Henceforward, the term "developer" applies to EnviroLogix.

The Developer, in consultation with ORNL, DOE, and EPA, is responsible for the following elements of this demonstration:

- Contribute to the design and preparation of the demonstration plan;
- Provide detailed procedures for using the technology;
- Prepare field-ready technology for demonstration;
- Operating and monitoring the technology during the demonstration;
- Documenting the methodology and operation of the technology during the demonstration;

- Furnish data in a format that can be compared to reference values;
- Logistical, and other support, as required.

ORNL has responsibilities for:

- Preparing the demonstration plan;
- Developing a quality assurance project plan (QAPP) (Section 8 of the demonstration plan);
- Preparing a health and safety plan (HASP) (Section 10 of the demonstration plan) for the demonstration activities;
- Developing a test plan for the demonstration;
- Acquiring the necessary reference analysis data;
- Performing sampling activities (including collecting, homogenizing, dividing into replicates, bottling, labeling, and distributing).

ORNL, DOE, and EPA have coordination and oversight responsibilities for:

- Providing needed logistical support, establishing a communication network, and scheduling and coordinating the activities of all demonstration participants;
- Auditing the on-site sampling activities;
- Managing, evaluating, interpreting, and reporting on data generated by the demonstration;
- Evaluating and reporting on the performance of the technologies.
- Site access:
- Characterization information for the site;
- Other logistical information and support needed to coordinate access to the site for the field portion of the demonstration, such as waste disposal.

3.0 TECHNOLOGY DESCRIPTION

This section provides a description of the EnviroLogix PCB in Soil Tube Kit. The description was provided by the technology developer, with minimal editing by ORNL. This section also describes that performance factors of the technology that will be assessed based on the data generated during the demonstration.

3.1 EnviroLogix PCB in Soil Tube Kit

3.1.1 Principle

The EnviroLogix PCB in Soil Tube Assay applies the principles of enzyme linked immunosorbent assay (ELISA) to the determination of PCB. In such an assay, an enzyme has been chemically linked to a PCB molecule or PCB analog to create a labeled PCB reagent. The labeled PCB reagent (called a conjugate) is mixed with an extract of native sample containing the PCB contaminant. A portion of the mixture is applied to a surface to which an antibody specific for PCB has been affixed. The native PCB and PCB-enzyme conjugate compete for a limited number of antibody sites. After a period of time, the solution is washed away, and what remains is either PCB-antibody complexes or enzyme-PCB-antibody complexes attached to the test surface. The proportion of the two complexes on the test surface is determined by the amount of native PCB in the original sample. The enzyme present on the test surface is used to catalyze a color change reaction in a solution added to the test surface. Because the amount of enzyme present is inversely proportional to the concentration of native PCB contaminant, the amount of color development is inversely proportional to the concentration of PCB contaminant. The color development is quantified through the use of a hand-held photometer.

The EnviroLogix PCB in Soil Tube Kit is designed for semi-quantitative field screening for PCBs in soil. The kit is supplied with calibrators equivalent to 1 part per million (ppm) and 10 ppm PCB (Aroclor 1254) in soil. These calibrators will be used to evaluate threshold levels of 1 and 10 ppm. If the sample is greater than 10 ppm, a threshold level of 50 ppm will also be evaluated using the 10 ppm calibrator by preparing a 1:5 sample extract dilution into methanol. For the extract samples, the threshold levels will be 0.4, 4, and $20 \mu g/mL$.

3.1.2 Applications and Advantages

The EnviroLogix PCB test kit can be used in a number of applications, including initial site characterization and mapping, real-time testing during remediation, and for screening negatives prior to GC confirmation. The test kit has a number of advantages:

- Real-time progress monitoring while crews and equipment are on-site
- Clear, accurate pass/fail determinations at meaningful action levels
- Meets site specific calibration needs without a special kit
- Reduces wastes and costs

3.1.3 Procedure

3.1.3.1 Materials

The EnviroLogix PCB in Soil Tube Kit contains the following items:

- 40 antibody-coated test tube
- 1 vial Negative Control
- 1 vial 1 ppm Calibrator
- 1 vial 10 ppm Calibrator
- 1 bottle of PCB-enzyme (horseradish peroxidase) Conjugate
- 1 bottle of Substrate
- 1 bottle of Stop Solution

The following items will need to be provided:

- EnviroLogix Soil Extraction Kit
- methanol (10 mL per sample)
- Repeater pipettes
- (3) 12.5 mL Combi-Syringes

- Positive displacement pipette
- marking pen
- timer
- distilled water
- portable photometer (Artel Differential Photometer or equivalent)
- test tube rack

3.1.3.2 Extraction

Weigh 5 g of soil into a soil extraction bottle containing two ball bearings. Add 10 mL of methanol and cap tightly. Shake vigorously by hand for 2 min. Let settle for 1 min. Pour extract into the base of the UniprepTM. Slowly push the filter plunger into the base until it stops at the bottom. To evaluate the samples relative to the 1 and 10 ppm calibrators, pour the filtered extracts into labeled 4 mL glass amber vials and cap tightly. To evaluate a 50 ppm threshold value, add 800 μ L of methanol to a 4 mL amber glass vial. Then add 200 μ L of the sample extract. This is a 1:5 dilution.

3.1.3.3 Assay

Allow all reagents to reach room temperature before beginning. Remove the number of antibody-coated test tubes (up to 20) necessary, and label one each for the negative control, the two calibrators, and the samples. Place the tubes in the test tube rack. Dispense $500~\mu L$ of conjugate into each tube, dispensing down the side of the tubes with the syringe tip at an angle to prevent splash back. Add $50~\mu L$ of sample to the appropriate tube(s). Then add $50~\mu L$ of negative control and calibrators to the appropriate tubes. Thoroughly mix the contents of the tubes by moving the rack in a rapid circular motion for 20-30 seconds. Let the tubes incubate for 10 min. Empty the tube contents into a suitable container. Fill the tubes with distilled water. Then empty them and shake out any remaining drops. Repeat the wash 3 times. Invert the tubes and tap them on paper towels to remove excess water. Add $500~\mu L$ of substrate to each tube. Thoroughly mix the contents of the tubes by moving the rack in a rapid circular motion for 20-30 seconds. Let the tubes incubate for 10 min. Note: If blue color does not develop in the negative control tube, the assay is invalid and should be repeated. Add $500~\mu L$ of stop solution to each tube. Read the tubes within 30 min of addition of the stop solution.

3.1.3.4 Interpreting Results

Use an Artel Differential Photometer (or equivalent) to measure the optical density of each tube's contents. The wavelength on the photometer should be set to 450 nanometers (nm). If the photometer has dual wavelength capability, use 600, 630, or 650 nm as the reference wavelength. If the photometer does not autozero on air, zero the instrument against 1 mL water in a blank tube. Measure and record the optical density (OD) of each tube's contents. Use Tables 3-1 and 3-2 to interpret the results.

The test kit results are reported as concentration ranges designated as intervals incorporating parentheses/bracket notation. The parentheses indicate that the end-points of the concentration range are excluded, while brackets indicate that the end-points are included. As shown in Table 3-1, the interval [0, 1) indicates that the PCB concentration range is greater than or equal to 0 and less than 1. If the sample is greater than 10 ppm, a 1:5 dilution of the sample will be prepared and assayed to determine if the concentration is greater than 50 ppm. This diluted sample will be evaluated using the 10 ppm calibrator, as shown in Table 3-2.

Table 3-1. Interpretation of photometer readings for undiluted samples

Samples with OD Values	Contain	And are reported as
> OD of 1 ppm calibrator	< 1 ppm PCB	[0, 1)
Between OD of 1 ppm and OD of 10 ppm calibrators	1 ≤ ppm PCB≤ 10	[1, 10]
< OD of 10 ppm calibrator	> 10 ppm PCB	(10, ∞)

Table 3-2. Interpretation of photometer readings for diluted samples

Diluted (1:5) Samples with OD Values	Contain	And are reported as
> OD of 10 ppm calibrator	10 < ppm PCB ≤ 50	(10, 50]
< OD of 10 ppm calibrator	> 50 ppm PCB	(50, ∞)

3.1.3.5 Precautions and Notes

The following items should be noted about the test kit:

- All components should be stored at 4° to 8° C when not in use. Allow reagents to come to room temperature before use. The components should not be used after the expiration date. It is important that the Substrate solution is not exposed to direct sunlight during pipetting or while incubating in the test tubes.
- The Stop Solution is 1.0 N hydrochloric acid and should be handled with caution.
- It is recommended that positive results be confirmed by an alternate method (such as gas chromatography).

3.1.4 Sensitivity and Cross-Reactivity

The test kit can be calibrated with other Aroclors. Table 3-3 shows the degree of sensitivity with the other Aroclors. It should also be noted that, at 1,000 ppm, the following compounds had low cross-reactivity (i.e., did not result in a positive response) at the 1 ppm interpretation level: 1,2-dichlorobenzene, 1,3-dichlorobenzene, 1,4-dichlorobenzene, 3,4-dichlorophenol, 2,5-dichlorophenol, biphenyl, pentachlorophenol, and humic acid.

Table 3-3. Sensitivity

Aroclor	Limit of Detection in Soil (ppm)
1242	1.7
1248	0.6
1254	0.3
1260	0.3

3.2 Demonstration Performance Goals

This section discusses the logistical and technical performances goals for the demonstration. Any method/instrument specification that is evaluated will be defensible by scientific data.

- Sample throughput
- Ease of use
- Completeness
- Blank results
- Precision
- Accuracy
- Comparability with reference laboratory results
- Application to regulatory-decision making (i.e., 50 ppm PCBs)
- Data quality

4.0 DEMONSTRATION SITE AND SAMPLE DESCRIPTIONS

This section discusses the history and characteristics of the demonstration site. The PCB samples to be analyzed are also described.

4.1 Site Name and Location

The demonstration of PCB field analytical technologies will be conducted at the Oak Ridge National Laboratory (ORNL) in Oak Ridge, Tennessee. PCB-contaminated soils from three DOE sites (Oak Ridge, TN, Paducah, KY, and Piketon, OH) will be used in this demonstration. The soil samples used in this study will be brought to the demonstration testing location for evaluation by EnviroLogix.

4.2 Site History

Oak Ridge, Tennessee, is located in the Tennessee River Valley, 25 miles northwest of Knoxville. Three Department of Energy (DOE) facilities are located in Oak Ridge: ORNL, the Y-12 plant, and the East Tennessee Technology Park (ETTP). Chemical processing and production of components for nuclear devices have occurred at the Y-12 Plant, and ETTP is a former gaseous diffusion uranium enrichment plant. At both facilities, industrial processing associated with nuclear weapons production has resulted in the production of millions of kilograms of PCB-contaminated soils. Two other DOE facilities—the Paducah plant in Paducah, Kentucky, and the Portsmouth plant in Piketon, Ohio—are also gaseous diffusion facilities with a history of PCB contamination. During the remediation of the PCB-contaminated areas at the three DOE sites, soils were excavated from the ground where the PCB contamination occurred, packaged in containers ranging in size from 55-gallon to 110-gallon drums, and stored as PCB waste. Samples from these repositories, referred to as "Oak Ridge", "Portsmouth", and "Paducah" samples, will be used in this demonstration.

In Oak Ridge, excavation activities occurred between 1991 and 1995. The Oak Ridge samples were comprised of PCB-contaminated soils from both Y-12 and ETTP. Five different sources of PCB contamination resulted in soil excavations from various dikes, drainage ditches, and catch basins. Some of the soils are EPA-listed hazardous waste due to the presence of other contaminants (e.g. diesel fuels).

A population of over 5,000 drums containing PCB-contaminated soils was generated from 1986 to 1987 during the remediation of the East Drainage Ditch at the Portsmouth Gaseous Diffusion Plant. The ditch was reported to have three primary sources of potential contamination: (1) treated effluent from a radioactive liquid treatment facility, (2) run-off from a biodegradation plot where waste oil and sludge were disposed, and (3) storm sewer discharges. In addition, waste oil was reportedly used for weed control in the ditch. Aside from PCB contamination, no other major hazardous contaminants were detected in these soils. As such, no EPA hazardous waste codes are assigned to this waste.

Twenty-nine drums of PCB-contaminated soils from the Paducah plant were generated as part of a spill cleanup activity at an organic waste storage area (C-746-R). The waste is considered a listed hazardous waste for spent solvents (EPA hazardous waste code F001) because it is known to contain trichloroethylene. Other volatile organic compounds, such as xylene, dichlorobenzene, and cresol, were also detected in the preliminary analyses of some of the Paducah samples.

4.3 Site Characteristics

Field demonstration activities will occur at two sites at ORNL: a natural outdoor environment (the outdoor site) and inside a controlled environmental atmosphere chamber (the chamber site). Figure 4-1 shows a schematic map of a section of ORNL indicating the demonstration area where the outdoor field activities will occur. Generally, the average September temperature for eastern Tennessee is 71°F. Studies will also be conducted inside a controlled environmental atmosphere chamber, hereafter referred to as the "chamber", located in Building 5507 at ORNL. The controlled experimental atmosphere facility consists of a room-size, walk-in chamber ten feet wide and twelve feet in length with air processing equipment to control temperature and humidity. Demonstration studies inside the chamber will be used to evaluate performance under environmental conditions that are markedly different from the ambient outdoor conditions at the time of the test. Generally, the temperatures in the chamber during the testing periods will be 55 to 60°F.

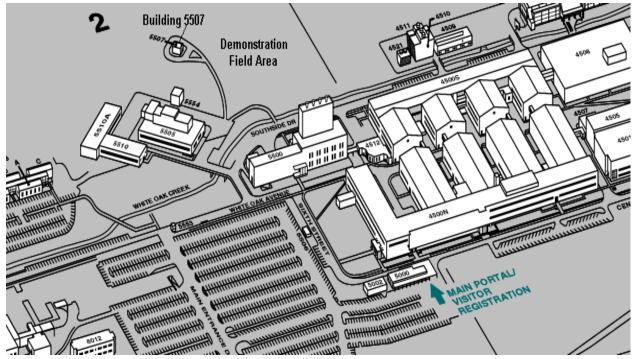


Figure 4-1. Schematic map of ORNL, indicating the demonstration area.

4.4 Soil Sample Descriptions

4.4.1 Oak Ridge Reservation, Portsmouth, and Paducah Soils

In Table 4-1 is presented a summary of the Oak Ridge Reservation, Portsmouth, and Paducah soils which will be evaluated as part of the PCB technology demonstration.

4.4.2 Tennessee Reference Soil

The soil is a Captina silt loam from Roane County, Tennessee that is slightly acidic (pH \sim 5) and low in organic carbons (\sim 1.5%). The soil composition is 7.7% sand, 29.8% clay, and 62.5% silt [1]. This soil will be used as the uncontaminated (blank) soil.

4.5 Extract Sample Descriptions

Traditionally, the amount of PCBs on a contaminated surface is determined by wiping the surface with a cotton pad saturated with hexane. The pad is then taken to the laboratory, extracted with additional hexane, and analyzed by gas chromatography. Unlike soil samples that can be more readily homogenized and divided, equivalent wipe samples (i.e. contaminated surfaces or post-wipe pads) were not easily obtainable. Therefore, interference-free solutions of PCBs prepared in methanol were analyzed to simulate an extracted surface wipe pad. Extract sample analyses provided evaluation data that primarily relied on the technology's performance rather than elements critical to the entire method (i.e. sample collection and preparation). A total of 12 extracts were analyzed per site; these consisted of four replicates each of a blank and two concentration levels (10 and $100 \mu g/mL$).

Table 4-1. Summary of Soil Sample Descriptions

Location	Request for Disposal (RFD) #	Drum #	Description
Oak Ridge	40022	02	Soil from spill cleanup at the Y-12 Plant in Oak Ridge, Tennessee. This soil is PCB-contaminated soil excavated in 1992.
Oak Ridge	40267	01 02 03 04	Soil from the Elza Gate area, a DOE Formerly Utilized Sites Remedial Action Program site in Oak Ridge, Tennessee. This soil is PCB-contaminated soil that was excavated in 1992.
Oak Ridge	24375	01 02 03	Catch-basin sediment from the K-711 area (old Powerhouse Area) at the DOE East Tennessee Technology Park (formerly known as Oak Ridge Gaseous Diffusion Plant) in Oak Ridge, Tennessee. This soil is PCB-contaminated storm drain sediment that was excavated in 1991.
Oak Ridge	43275	01 02	Soil from the K-25 Building area at the DOE East Tennessee Technology Park (formerly known as Oak Ridge Gaseous Diffusion Plant) in Oak Ridge, Tennessee. This soil is PCB-contaminated soil that was excavated in 1993.
Oak Ridge	134555	03	Soil from the K-707 area at the DOE East Tennessee Technology Park (formerly known as Oak Ridge Gaseous Diffusion Plant) in Oak Ridge, Tennessee. This soil is PCB-contaminated soil from a dike spillage that was excavated in 1995.
Paducah	97002	01 02 03 04	Soil from the DOE Paducah Gaseous Diffusion Plant in Kentucky. This soil is PCB-contaminated soil from a spill cleanup at the C-746-R (Organic Waste Storage Area) that was excavated in 1989.
Portsmouth	7515	858 1069 1096 1898 2143 2528 3281 538 940 4096	Soil from the DOE Portsmouth Gaseous Diffusion Plant in Ohio. This soil is PCB-contaminated soil from a probable PCB oil spill into the East Drainage Ditch that was excavated in 1986.

4.6 Sample Stability Study

EnviroLogix will be demonstrating their immunoassay technology using the same samples that were used in the July 1997 demonstration of six PCB technologies. Soil samples are available for the EnviroLogix demonstration because extra samples were prepared and stored since 1997. ORNL performed chemical analyses of representative samples to verify that significant amounts of PCBs had not been lost due to storage for one year. Duplicate analyses from each unique soil sample were performed. It was confirmed that no considerable losses in PCB concentration had occurred, and therefore, all soil samples could be utilized in the EnviroLogix demonstration. The extract samples were also archived since the 1997 demonstration. However, because these samples were prepared in methanol, it was assumed that these samples would have degraded in a year. Therefore, new extract samples will be prepared by ORNL. For these samples, the EnviroLogix's result will be compared to the nominal concentration value only, instead of the reference laboratory result.

5.0 CONFIRMATORY PROCESS

The verification process is based on the presence of a statistically validated data set against which the performance goals of the technology may be compared. The choice of an appropriate reference method and reference laboratory are critical to the success of the demonstration.

5.1 Method Selection

The reference analytical method will be EPA SW-846 Method 8081 [2].

5.2 Reference Laboratory Selection

To assess the performance of the PCB field analytical technology, the data obtained using the technology will be compared to data obtained using conventional analytical methods. This decision is based on the experience of prospective laboratories with QA procedures, reporting requirements, and data quality parameters consistent with the goals of the Program. The laboratory must also demonstrate past proficiency with the method.

At the time of the 1997 demonstration, Oak Ridge Sample Management Office (SMO) was tasked by DOE Oak Ridge Operations with maintaining a list of qualified laboratories to provide analytical services. In Appendix A are presented the standard operating procedures that SMO used to identify, qualify, and select analytical laboratories. The first procedure (LMES-ASO-AP-203, REV. 0) describes the process for selecting, adding and expelling commercial laboratories to the LMES Pricing Agreement. The second procedure (LMES-ASO-AP-210, REV. 0) defines the methodology used by Oak Ridge Sample Management Office personnel in processing statements of work (SOWs), processing purchase requisitions, and selecting commercial analytical laboratories. These activities for the procurement of commercial laboratory services were used to support projects sponsored by the DOE Oak Ridge Operations Office. The procedure served to ensure that as an operation of a DOE contractor, LMES SMO maintained an optimum level of technical and administrative oversight on each project, and SMO commercial procurement activities complied with federal acquisition laws and LMES procurement policy.

Using the procedures listed in Appendix A, ORNL and SMO selected LAS Laboratories, in Las Vegas, NV, as the reference laboratory. In Appendix B is presented the LAS standard operating procedure.

5.3 In-Field Support Laboratory

ORNL-based Grand Junction, Colorado (ORNL-GJ) field team served as the in-field support laboratory for the preliminary on-site analyses of the PCB-contaminated soils. In Appendix C is presented ORNL-GJ's analytical procedures. ORNL's Chemical and Analytical Sciences Division (CASD) also performed preliminary characterization of the PCB-contaminated soils using a similar procedure.

5.4 Special QC Requirements

In order to increase the likelihood that high quality data will be obtained, an enhanced QC strategy will be required. Standard reference materials, double blind standards, matrix spiked soils, and special performance evaluation materials will be utilized.

5.5 Laboratory Audit

The SMO conducts on-site audits of LAS annually as part of the laboratory qualification program. At the time of selection, the most recent audit of LAS had occurred in February 1997. Results from this audit indicated that LAS was proficient in several areas, including program management, quality management, and training programs. No findings regarding PCB analytical procedure implementation were noted. A second on-site assessment of LAS occurred August 11–12, 1997, during the analysis of the demonstration study samples.

This surveillance focused specifically on the procedures that were currently in use for the analysis of the demonstration samples. The audit, jointly conducted by the SMO, DOE-ORO, and EPA ESD-LV, verified that LAS was procedurally compliant. The audit team noted that LAS had excellent adherence to the analytical protocols and that the staff were knowledgeable of the requirements of the method. No findings impacting data quality were noted in the audit report.

6.0 PCB SOIL SAMPLE COLLECTION

6.1 Sample Collection Plan

In Appendix D is presented the sample collection plan. The sample collection plan for this demonstration specifies the procedures that were used to ensure the consistency and integrity of the samples. In addition, this plan outlines the sample collection procedures necessary to meet the demonstration purpose and objectives.

6.1.1 Sample Collection Procedures

Sampling occurred at the K-25 site for several days over the period of April 17 through May 7, 1997. Portsmouth and Oak Ridge Reservation soils were collected from B-25 storage boxes and from 55-gallon drums. Figure 6-1 is a photo of the Analytical Services Organization's sampling team acquiring some PCB soil samples from a 55-gallon drum.

Soil was collected from the top of the drum and placed in a plastic bag. The soil was then sifted by hand to remove rocks and other large debris, and placed in a plastic-lined 5-gallon container. Figure 6-2 shows the samplers performing this procedure. The amount of soil collected half-filled the 5-gallon container, amounting to approximately 12 kg of soil.



FIGURE 6-1: K-25 personnel acquire a PCB soil sample from a 55-gallon drum.



FIGURE 6-2: K-25 sampling personnel sift through the collected soil to remove rocks and other large debris.

Once the sifting was completed, the plastic liner was then removed from the container. To homogenize the soil sample, the liner was rolled on the ground in a back and forth motion, such the sample was kneaded and thoroughly mixed. Two 40-mL amber vials were fill with the homogenized soil for preliminary analytical characterization. A third sample was taken for total radiological activity screening. Paducah soil samples were collected at the site and shipped to ORNL for use in the demonstration.

6.2 Preliminary Soil Characterization

The two analytical samples taken of the homogenized soil were analyzed using the procedure described in Appendix C. The analyses were performed by ORNL-GJ and ORNL/CASD. The total PCB concentration was measured in each analytical sample to determine which samples would be used in the demonstration. Results from the total activity screening indicated that the soils were not considered radioactive.

6.3 Predemonstration Sample Preparation, Distribution, and Analysis

A predemonstration sampling and analysis event is required to allow the technology developers to refine their technologies and revise their operating instructions, if necessary. This analysis also allows an evaluation of matrix effects or interferences that may affect the demonstration. A failure to meet the performance goals at this point could indicate a lack of maturity of the technology and the demonstration would be canceled.

This sampling requirement has the following objectives:

- To allow the developers to analyze samples that will be included in the demonstration in advance, and, if necessary, refine and calibrate their technologies and revise their operating instructions
- To allow an evaluation of any unanticipated matrix effects or interferences that may occur during the demonstration

For the predemonstration study, EnviroLogix will analyze five performance evaluation (PE) soils and one extract sample. Pre-prepared certified PE samples were obtained from Environmental Resource Associates (ERA) and EPA's Office of Solid Waste and Emergency Response's Analytical Operations and Data Quality Center. The soils purchased from ERA (Arvada, CO) had been prepared using ERA's semivolatile blank soil matrix. This matrix was a top soil that had been dried, sieved, and homogenized. Particle size was

approximately 60 mesh. The soil was approximately 40% clay. Samples acquired from the EPA's Office of Solid Waste and Emergency Response's Analytical Operations and Data Quality Center had been prepared using contaminated soils from various sites around the country in the following manner: The original soils had been homogenized and diluted with a synthetic soil matrix (SSM). The SSM had a known matrix of 6% gravel, 31% sand, and 43% silt/clay; the remaining 20% was top soil. The dilution of the original soils was performed by mixing known amounts of contaminated soil with the SSM in a blender for no less than 12 hours. The samples were also spiked with target pesticides (α , β , Δ , and δ -BHC, methoxychlor, and endrin ketone) to introduce some compounds that were likely to be present in an actual environmental soil. The hydrocarbon background from the original sample and the spiked pesticides produced a challenging matrix. Additionally, ORNL will prepare a solvent extract in methanol at one of the two concentration levels that will be evaluated during the demonstration.

6.3.1 Predemonstration Sample Distribution

The predemonstration samples will be sent to the developer on August 10, 1998. In Appendix E are presented the pre-demonstration study instructions. The test kit results for the predemonstration sample analyses will be provided to ORNL approximately one week after the receipt of the samples (August 18, 1998).

6.3.2 Predemonstration Sample Analysis Results

For the PE soils, the developer predemonstration results will be compared to the reference laboratory results. Additionally, the results will be compared to performance acceptance ranges. The acceptance ranges, based on the analytical verification data, are guidelines established by the provider of the PE materials to gauge acceptable analytical results. For the extract sample, the test kit result will be compared to the nominal concentration. The PCB in Soil Tube Assay kit's performance on these samples will determine if the technology is mature and ready for field testing.

6.4 Sample Preparation for Demonstration

The PCB soil samples were homogenized (dried, sieved, and thoroughly mixed) prior to sample splitting. Each sample, contained in 4 ounce glass jars, consists of approximately 20 g of sample. The extract samples will be prepared in methanol. The extracts will be stored in the refrigerator (≤ 4 °C) until released to the developer.

The environmental soil samples were characterized in terms of composition (% sand, % gravel, % silt/clay, etc.), total organic carbon, and pH. This data will be reported in the technology verification report.

6.5 Sample Labeling for Demonstration

The samples are labeled with a PCB warning label. Each jar is also labeled with a sample number. Replicate samples are assigned unique (but not sequential) sample numbers. PE materials are labeled in the same manner, such that the PE samples are indistinguishable from other samples. The order of analysis will be randomized and set for the developer.

7.0 DEMONSTRATION DESIGN

This section discusses the objectives of the demonstration, factors that must be considered to meet the performance objectives, and the information that ORNL, DOE, and EPA will use to evaluate the results of the demonstration.

7.1 Objectives

The primary objectives of this demonstration are to evaluate the PCB field analytical technologies in the following areas: (1) how well each performs relative to conventional analytical methods, (2) the impacts of sample matrix variations on performance, (3) the affect that environmental conditions have on performance, (4) PE results, and (5) the logistical and economic resources necessary to operate the technology. Secondary objectives for this demonstration are to evaluate the PCB field analytical technology in terms of its reliability, ruggedness, cost, range of usefulness, sample throughput, data quality, and ease of operation. Where possible, the performance will be compared to the performance of conventional analytical methods used in performing similar site characterization activities. The verification process will also evaluate the performance of the technology against the performance goals as stated in Section 3.2.

7.2 Experimental Performance Measures

This section discusses performance measures that will be considered in the design and implementation of the demonstration. These performance measures include accuracy, precision, portability, ease of operation, ruggedness, health and safety issues, sample throughput, and sample matrix effects.

7.2.1 Qualitative Performance measures

Some performance measures, while important, are difficult or impossible to quantify. These are considered qualitative performance measures: ease of operation, operator training requirements, portability, ruggedness, and special requirements.

7.2.2 Quantitative Performance measures

Many performance measures in this demonstration can be quantified by various means, including the following: accuracy, precision, number of false positive (fp) results, number of false negative (fn) results, waste generation, affect of environmental conditions on operation (controlled environmental atmosphere studies), sample throughput, and operating costs. These quantitative performance measures will be used to assess the technology performance by comparison to reference laboratory data, where possible.

Another objective of this demonstration is to assess the technology's ability to perform at regulatory decision-making levels for PCBs, specifically 50 ppm for soils and $100~\mu g/100 cm^2$ for surface wipes. To assess this ability for soils, the test kit's performance for PE and environmental soil samples ranging in concentration from 40 to 60 ppm (as determined by the paired reference laboratory analyses) will be used. For this concentration range, the percentage of test kit results that agree with, are above (i.e., biased high), and are below (i.e., biased low) the reference laboratory results will be reported. Due to the limited number of extract samples, all sample results will be considered for this assessment.

7.3 Experimental Factors

7.3.1 Glossary of Terms

The experimental factors were selected to represent field conditions. The experimental design afforded an examination of the the effect of environmental conditions, concentration levels, and soil matrix types on the PCB measurements.

<u>Chamber</u> - room-size controlled environmental atmosphere facility at ORNL. The developers will demonstrate their technologies inside the chamber under temperature and relative humidity conditions that are different from the ambient conditions. The chamber will be set at $55^{\circ}F$ and 50% relative humidity. This will be a cost effective approach to simulate demonstrating the technologies at a second site.

PE sample - certified soil sample containing known concentrations of PCBs. The soils will consist of ones

purchased from Environmental Resource Associates and obtained from the U. S. EPA's Office of Solid Waste and Emergency Response's Analytical Operations Center.

<u>Reference Laboratory</u> - an analytical laboratory that perform ed EPA SW-846 (Method 8081) analyses of the PCB samples for comparison with developer field results. LAS Laboratories (Las Vegas, NV) was the reference laboratory.

<u>Outdoor site</u> - area west of Building 5507 at Oak Ridge National Laboratory. During the demonstration, temperature and relative humidity conditions will be recorded. The average temperature for September in eastern Tennessee is 71°F.

<u>Chamber site</u> - controlled environmental atmosphere facility located in Building 5507 at Oak Ridge National Laboratory. The chamber settings will be 55° F and 50% relative humidity.

<u>Environmental Soil</u> - an environmental soil sample collected from Oak Ridge, Paducah, and Portsmouth sites. These samples will range from more simple, single Aroclor samples to more challenging mixtures of Aroclors with high oil and hydrocarbon contamination.

Spike - an environmental soil sample that has been spiked with additional amounts of PCBs

<u>Extract samples</u> - a methanol extract containing known concentrations of PCBs. This will simulate a surface wipe sample that was collected and extracted.

7.3.2 Summary of Demonstration Activities

The demonstration is scheduled to be held at ORNL from September 21 through September 25, 1998. The soil samples evaluated during the demonstration consist of (1) environmental soil samples from the Oak Ridge Reservation, Paducah, and Portsmouth DOE sites, (2) spiked environmental soil samples, (3) purchased certified soil samples, and (4) ORNL-prepared methanol extract samples. The demonstration soil samples has been homogenized and split such that the developer is supplied with equivalent samples that have been analyzed by a fixed analytical laboratory (referred to as the reference lab). The test kit results for the extract samples will be compared to the nominal spike concentration. Some features of the approach are presented in Table 7-1. The experimental design approach is presented in Tables 7-2 through 7-5. The developer will analyze a total of 232 samples in all. At each of the two sites, 36 PE samples, 68 environmental soils, and 12 extract samples will be analyzed.

Table 7-1. Experimental Design Features

Properties: 17 unique samples per site; acquire more data on fewer samples; statistically rich approach

Replicates: equal number (quadruplicate) for all soil types, extract samples, and concentration levels

Accuracy: equal number of comparisons with certified and spike concentrations for the PE soils and extract samples, respectively, at all concentration levels

Precision: estimated for all soil types, extract samples, and concentration levels

Data Analysis: simplified statistics due to consistency with number of replicates

Table 7-2. Summary of Environmental Soil Sample Analyses (by Drum Number)

Target	Outdoor Site				Chamber Site			
Concentration Range	Oak Ridge#1	Oak Ridge#2	Paducah#1	Totals # Samples	Paducah#1	Portsmouth#1	Portsmouth#2	Total # Samples
0.1 - 2.0 ppm	40022-02 a	24375-01 40267-02 24375-02	97002-04 97002-01	28	97002-04 97002-01	7515-4096		12
2.1 - 20.0 ppm	40267-03 40267-01 40267-04		97002-03	16	97002-03	7515-1898	7515-2528 7515-3281	20
20.1 - 50.0 ppm		134555-03S	97002-02	12	97002-02	7515-1096 7515-2143	7515-1069 7515-0858	24
50.1 - 700 ppm	40267-01S ^b 24375-03	43275-01 43275-02	97002-02S	12	97002-02S	7515-0940 7515-0538S	7515-0538 7515-0538S	12
Total # samples	24	24	20	68	24	24	20	68
Grand Total								136

<sup>Four replicates will be analyzed for each drum number.
"S" indicates that the sample is a matrix spiked environmental sample.</sup>

Table 7-3. Summary of Performance Evaluation Soil Samples

Tuole 7 et Summing of 1 errormance 27 actualism		Number of Replicates		
Sample	Concentration (ppm)	Outdoor Site	Chamber Site	
A 1 1040 9	2	4	4	
Aroclor 1248 a	20	4	4	
1 10710	5	4	4	
Aroclor 1254 ^a	50	4	4	
A 12.0 8	11	4	4	
Aroclor 1260 a	50	4	4	
M'	2 °	4	4	
Mixture of Aroclor 1254 and 1260 b	50 °	4	4	
Uncontaminated (blank) soil (Tennessee Reference Soil)	n/a	4	4	
Total # samples	36	36		
Grand Total		72		

^a Provided by the EPA's Office of Solid Waste and Emergency Response's Analytical Operations and Data Quality Center.

^b Provided by Environmental Resource Associates.

^c Total PCB concentration

Table 7-4. Summary of Extract Sample Analyses

	Number of			
Sample Concentration	Outdoor Site	Chamber Site	Grand Total	
10 μg/mL	4	4	8	
100 μg/mL	4	4	8	
Methanol Blank	4	4	8	
Total # samples	12	12	24	

Table 7-5. Summary of Demonstration Analyses

C I T	Number of analyses			
Sample Type	Outdoor Site	Chamber Site		
Environmental samples	68	68		
PE samples	36	36		
Extract samples	12	12		
Grand Totals	116	116		

7.4 Field Data

The technology will be operated by the developer, who will provide the results to ORNL. The developer will be responsible for reducing the raw data into a presentation format consistent with the evaluation requirements. At the end of the demonstration, the developer will submit all final results and raw data to ORNL.

7.4.1 Field Audit

The EPA, DOE, and ORNL will conduct audits of all field activities. This activity will document any deviations from the demonstration plan, operational details, and other performance measures associated with the evaluation of the field technology. Audit notes will be included as part of the Quality Assurance Project Plan (Section 8.0).

7.5 Demonstration Schedule

Demonstration activities will occur from September 21 through September 25, 1998. Visitors will be invited to talk with EnviroLogix and view technology demonstrations on the morning of September 16.

7.6 Field Operations

This demonstration requires close communication between the developer, ORNL, DOE, and EPA. Preliminary site training (on September 21) will be required before initiation of field study. Successful field operations require detailed planning and extensive communication. The implementation of the demonstration must be consistent with the requirements of the study and routine operation of the technology.

7.6.1 Communication and Documentation

ORNL will communicate regularly with the demonstration participants to coordinate all field activities associated with this demonstration and to resolve any logistical, technical, or QA issues that may arise as the demonstration progresses. The successful implementation of the demonstration will require detailed coordination and constant communication between all demonstration participants. All developer/ORNL field activities will be thoroughly documented. Field documentation will include field logbooks, photographs, field data sheets, and chain-of-custody forms.

The ORNL field team leader will be responsible for maintaining all field documentation. Field notes will be kept in a bound logbook. Each page will be sequentially numbered and labeled with the project name and number. Completed pages will be signed and dated by the individual responsible for the entries. Errors will have one line drawn through them and this line will be initialed and dated. Any deviations from the approved final demonstration plan will be thoroughly documented in the field logbook and provided to the ORNL. Photographs will be taken with a digital camera.

The developer will obtain all equipment needed for field work associated with this demonstration. Prior to the demonstration, EnviroLogix will work with ORNL to secure any equipment requirements (such as tables, chairs, etc.) that the developer will need for the demonstration.

7.6.2 Sample Distribution

ORNL will be responsible for sample distribution. The samples will be packaged in 4 ounce (120 mL) jars, as described in Section 6.4. All samples will be prepared for distribution at the start of the demonstration. EnviroLogix will go to a sample distribution table located in Building 5507 to pick-up the samples. The samples will be distributed in batches of 12. Completion of chains-of-custody will document sample transfer.

7.6.2.1 Archive Samples

Archive samples which are replicates of the developer samples will be retained by ORNL. An archive

sample will be used during the demonstration if the integrity of a developer's sample has been compromised. Additional unhomogenized material and unused archive samples will also be retained at ORNL at the completion of the demonstration, in case any questions arise where reanalysis is necessary.

7.7 Statistical Analysis of Results

The performance of EnviroLogix's PCB in Soil Tube Assay will be evaluated using precision, accuracy, representativeness, completeness, and comparability (PARCC) parameters [3], which are indicators of data quality. Results will be evaluated from the analysis of PE, environmental soil, and extract samples. The PARCC parameters will be defined in the following manner:

- Precision: Precision is the reproducibility of measurements under a given set of conditions. The frequency with which the same interval is reported within a set of replicates will be used to quantify precision. Reporting a higher number of replicates in the same interval for a given replicate set will indicate higher precision. In other words, reporting all four replicate results as the same interval will indicate the highest possible precision.
- Accuracy: Accuracy represents the closeness of the test kit's measured PCB concentrations to the certified values. Because the test kit produces interval results, accuracy will be evaluated in terms of the percentage of samples which agree with, are above (i.e., biased high), and are below the certified value (i.e., biased low). Accuracy will also be assessed by the number of false positive and false negative results that are produced by the kit. A false positive (fp) result [4] is one in which the technology detects PCBs in the sample when there actually are none. A false negative (fn) result [4] is one in which the technology indicates that there are no PCBs present in the sample, when there actually are. Both fp and fn results are influenced by the method detection limit of the technology.
- Representativeness: Representativeness expresses the degree to which the sample data accurately and precisely represent the capability of the technology. The performance data will be accepted as representative of the technology if the test kit is capable of analyzing diverse samples types (PE samples, simulated wipe extract samples, and actual field environmental samples) under multiple environmental conditions.
- Completeness: Completeness is defined as the percentage of measurements that are judged to be useable (i.e., the result was not rejected). The optimum completeness is 95% or greater.
- Comparability: Comparability refers to the confidence with which one data set can be compared to another. A one-to-one sample comparison of the test kit results and the reference laboratory results will be performed for all samples. Similar to accuracy, the test kit results will be evaluated in terms of the percentage of samples which agree with, are above (i.e., biased high), and are below (i.e., biased low) relative to the results generated by the reference laboratory.

8.0 QUALITY ASSURANCE PROJECT PLAN (QAPP)

The QAPP for this demonstration specifies procedures that will be used to ensure data quality and integrity. Careful adherence to these procedures will ensure that data generated from the demonstration will meet the desired performance objectives and will provide sound analytical results.

8.1 Purpose and Scope

The primary purpose of this section is to outline steps that will be taken by the developer to ensure that data resulting from this demonstration is of known quality and that a sufficient number of critical measurements are taken. EPA considers the demonstration to be classified as a Category II project. This section of the demonstration plan addresses the key elements that are required for Category II projects prepared according to guidelines in the EPA guidance documents "Preparation Aids for the Development of Category II Quality Assurance Project Plans" (Simes 1991), "Preparing Perfect Project Plans (1989), and the Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans" (Stanley and Verner 1983).

8.2 Quality Assurance Responsibilities

The developer project manager is responsible for coordinating the preparation of the QAPP for this demonstration and for its approval by the EPA project manager and ORNL. The developer project manager will ensure that the QAPP is implemented during all demonstration activities. The developer QA manager for the demonstration will review and approve the QAPP and will provide QA oversight of all demonstration activities. The QA audit function will be the responsibility of the EPA.

Samples will be analyzed on site by the PCB field analytical technology and off site by the reference laboratory using EPA SW-846 Method 8081. Primary responsibility for ensuring that activities comply with the requirements of the demonstration will rest with the EPA technical lead and ORNL technical lead. QA/QC activities for the PCB field analytical technology will include those activities recommended by developer and those required by the EPA or ORNL to assure the demonstration will provide data of the necessary quality.

8.3 Data Quality Indicators

The data obtained during the demonstration must be of sufficient quality for conclusions to be drawn on the PCB field analytical technology. For all measurement and monitoring activities conducted for EPA, the Agency requires that data quality parameters be established based on the proposed end uses of the data. Data quality parameters include five indicators of data quality: representativeness, completeness, comparability, accuracy, and precision.

Data generated by the PCB field analytical technology will be compared to the data generated from LAS Laboratories. High quality, well documented reference laboratory results are essential for meeting the purpose and objectives of this demonstration. LAS Laboratories data has been validated by ORNL for comparison with the technology developer data. The following indicators of data quality will be closely evaluated to determine the performance of the technology when measured against data generated by the reference laboratory.

8.3.1 Representativeness

Representative samples, in general, are samples that contain a reasonable cross-section of the "population" over which they are to be used to make inferences. The population for demonstrations analyzed as part of this project includes a variety of media and contaminants that the innovative technologies are developed to accommodate.

This demonstration will evaluate the technology under multiple conditions, while leveraging resources by: (1) conducting the demonstration at one site and utilizing a controlled environmental atmosphere to simulate temperature and humidity conditions in another part of the country; (2) evaluating PCB-contaminated soil samples from three different DOE sites, namely Portsmouth, Paducah, and the Oak Ridge Reservation; and (3) studying a wide range of PCB concentrations (0 to 700 ppm).

8.3.2 Comparability

Comparability is a quality parameter determined for the most part in the planning stages of the demonstration, often on the basis of prior knowledge of the innovative technologies' performance capabilities. First, the innovative technology must be comparable in some way to a reference or baseline method for the demonstration to be worthwhile. The study has been designed such that it is a statistically-rich approach that allows for an equal number of comparisons for every soil type and concentration level. Therefore, direct comparisons can be made with the reference laboratory results. However, enough replicates and quality control samples will be analyzed to independently assess each technology's performance.

8.3.3 Completeness

Completeness refers to the amount of data collected from a measurement process expressed as a percentage of the data that would be obtained using an ideal process under ideal conditions. The completeness objective for data generated during this demonstration is 95% or better.

There are many instances which might cause the sample analysis to be incomplete. Some of these are:

- ► Instrument failure
- ► Calibration requirements not being met
- Evaluated analyte levels in the method blank

8.3.4 Accuracy

Accuracy is a measure of how close, on average, values of the innovative technology are to the true values. Inaccuracies or biases are the result of systematic differences between these values. When comparing the innovative technology to a reference technology difficulties can arise. In some cases biases can be attributed to the innovative technology. These biases are often the result of poor calibration. Other possible sources of bias include systematic errors in standards preparation, biases introduced in the sample extraction, storage and shipping processes and biases resulting from setup-related differences at the reference laboratory. Only the former of these sources is likely to be incurred by users of the innovative technologies. Most of the remaining sources represent inaccuracy that might be avoided through use of the innovative technology. Consequently every effort should be made by ORNL, the developer, and the reference laboratory to identify specific sources of bias. The design of blanks, replicates and performance assessment samples should provide substantiating evidence to support this partitioning of sources of inaccuracy when results become available.

The strength of this demonstration's experimental design is that since an equal number of replicates will be performed for every samples at every concentration level, an equal number of accuracy comparisons can be made. However, enough replicates and quality control samples will be analyzed to independently assess each technology's performance.

8.3.5 Precision

Precision, in general, refers to the degree of mutual agreement among measurements of the same materials and contaminants. Environmental applications often involve situations where "measurements of the same materials" can take on a number of interpretations. In environmental applications, precision is often best specified as a percentage of contaminant concentration. The following lists several possible interpretations of precision for environmental applications.

- 1) The precision involved in repeated measurements of the same sample without adjusting the test equipment.
- 2) The precision involved in repeated measurements of the same sample after reset, repositioning, or re-calibration of the test equipment or when using different equipment of the same technology.
- 3) The precision of measurements due to spatial variability of soil samples from adjacent locations.
- 4) The precision characteristics of a specific technology in determining contamination at a specific site or at an arbitrary site.

In general, users of the technology will want to be assured that measurement variability in 1) and 2) is small. Measurement variability due to spatial variability described in 3) is likely to be site specific and is minimized in this demonstration by using homogeneous samples. The measurement variability discussed in 4) is perhaps of most interest as it includes measurement variability resulting from possible differences in the design activities and effects of environmental conditions such as temperature that would vary from one site characterization to another as well as site and technology specific sources.

The strength of this demonstration's experimental design is that since an equal number of replicates will be performed for every sample at every concentration level, an equal number of precision comparisons can be made. However, enough replicates and quality control samples will be analyzed to independently assess each technology's performance.

8.4 Calibration Procedures and Quality Control Checks

This section describes the calibration procedures and method-specific QC requirements that apply to both the technology and the reference analyses. It also contains a discussion of the corrective action to be taken if the QC parameters fall outside of the evaluation criteria.

8.4.1 Initial Calibration Procedures

Initial calibration for the technology will be performed according to the developer's recommendation (see technology descriptions, Section 3.0). The reference laboratory's initial calibration procedure is described in Appendix B.

8.4.2 Continuing Calibration Procedures

Continuing calibration for each technology will be performed according to the developer's recommendation (see technology descriptions, Section 3.0). The reference laboratory's continuing calibration procedure is described in Appendix B.

8.4.3 Method Blanks

A method blank is an analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing, and is carried through the complete sample preparation and analytical procedures. Four method blanks will be included as part of the PE/QC program (see Table 7-3).

8.4.4 Spike Samples

The spiked soil samples used in this demonstration will be matrix spiked environmental samples. To prepare a spiked sample, the soil is first ground either using a mortar and pestle or a conventional blender.

(Real samples will be oven-dried prior to grinding.) The soil is then sieved through a screen which was 16 mesh, or 1 mm particle size. The sieved soil is spiked with a diethyl ether solution of PCBs at the desired concentration. The soil is agitated using a mechanical shaker, then allowed to air-dry overnight. Several spiked samples are incorporated into the experimental design (see Table 7-2).

LAS Laboratories also prepared and analyzed matrix spike /matrix spike duplicate samples (MS/MSD) samples with every analytical batch. (The analytical batch can include no more than twenty samples, excluding blanks, standards, spikes, and dilutions.) Aroclor 1260 was the matrix spike analyte.

8.4.5 Laboratory Control Samples

Laboratory control samples are samples of known composition that are analyzed periodically to assure that the analytical system is in control. These are analyzed just like a regular sample. One LCS was analyzed per analytical batch. LAS used purchased certified LCS standards.

8.4.6 Performance Evaluation Materials

The certified concentrations of the PE samples will be used to evaluate the PCB field analytical technology. The PCB field analytical technology will analyze the PE samples periodically during the demonstration. PE samples will be obtained from Environmental Resource Associates and the U. S. EPA's Office of Solid Waste and Emergency Response's Analytical Operations and Data Quality Center.

8.4.7 Replicate Samples

All of the samples (real, PE/QC, blank, extracts) will be analyzed in quadruplicate so that the precision of the technology can be determined independently and compared.

8.5 Data Reduction, Review, and Reporting

To maintain good data quality, specific procedures will be followed during data reduction, review, and reporting. These procedures are detailed below.

8.5.1 Data Reduction

Data reduction refers to the process of converting the raw results from the technology into a concentration or other data format which will be used in the comparison. The procedures to be used will be technology dependent, but the final result format will be comparable to the reference lab results. The actual comparisons will be performed by ORNL. The following is required for data reduction:

<u>Concentrations</u>: The report PCB concentration should be total PCB concentration in parts per million (i.e., ppm, as received) for soil samples and μ g/mL for extract samples. (See Tables 3-1 and 3-2 for more information.)

Nondetect Concentrations: If no PCB is detected, the concentration should be reported as [0, 1) ppm.

8.5.2 Data Review

The developer will verify the completeness of the appropriate data forms and the completeness and correctness of data acquisition and reduction. The independent technology observer will review calculations and inspect laboratory logbooks and data sheets to verify accuracy, completeness, and adherence to the specific analytical method protocols. Calibration and QC data will be examined by the individual developers and DOE, EPA, and ORNL observers. The individual developers will verify that all instrument systems are in control and that QA objectives for accuracy, completeness, and method detection limits have been met.

8.5.3 Data Reporting

This section contains a list of the data to be reported by both the technology and the reference method. At a minimum, the data tabulation will list the results for each sample and include reporting units, sample numbers, results, and data qualifiers. (A sample results form will be provided for completion by the developers.) All QC information such as calibrations, blanks and reference samples will also be included with the raw analytical data. All data should be reported in hardcopy.

Developer results will be due to ORNL at the conclusion of a day's field activities. The developer's final report will be due to ORNL one week after the conclusion of the demonstration. Any discrepancies between the originally reported result and the final result must be described.

8.6 Calculation of Data Quality Indicators

Precision, in general, refers to the degree of mutual agreement among measurements of the same materials and contaminants. Precision for the PCB verification demonstration will be estimated by the variance, or standard deviation from the measured data. If "n" PCB concentration measurements are represented by $Y_1, Y_2, ..., Y_n$, the estimated variance about their average value " $\overline{\gamma}$ " is calculated by:

$$S^2 = \frac{1}{n-1} \sum_{k=1}^{n} (Y_k - \overline{Y})^2$$
.

The standard deviation is the square root of S^2 and implies that the uncertainty is independent of the PCB concentration values. To express the reproducibility relative to the average PCB concentration, percent relative standard deviation (RSD) is used to quantify precision, according to the following equation:

$$RSD = \frac{Standard\ Deviation}{Average\ Concentration} \ x\ 100\%$$

Replicate samples at each PCB concentration can be used to establish the relationship between the uncertainty and the average PCB concentration. RSD cannot be calculated for PCB concentration results reported as interval data, which is how EnviroLogix's test kit data is reported. To assess precision, the frequency of results reported as the same interval will be determined.

Accuracy is a measure of how close, on average, the measured PCB concentrations are to the true values or to an accepted reference value. Accuracy for the PCB verification demonstration will be relative to a standard PCB concentration in the case of performance evaluation samples or to a reference value measured by a reference laboratory.

$$percent \ recovery = \frac{measured \ amount}{actual \ amount} \ x \ 100\%$$

The optimum percent recovery value is 100%. Percent recovery values greater than 100% indicate results that are biased high, and values less than 100% indicate results that are biased low. Percent recovery will be used

to assess the accuracy of the reference laboratory measurements relative to the certified PE concentrations (or the spiked concentrations for the extract samples). Because the test kit produces interval results, accuracy will be evaluated in terms of the percentage of samples which agree with, are above (i.e., biased high), and are below the certified values (i.e., biased low).

Inaccuracies or biases are the result of systematic differences between measured and true values. These biases may be due to limited calibration range, systematic errors, standards preparation, storage and homogeneity of the soil samples either at the PCB verification demonstration or at the reference laboratory. Consequently every effort will be made by ORNL, the technology developers and the reference laboratory to identify specific sources of inaccuracies. The demonstration includes blanks, replicates, and performance evaluation samples that should provide substantiating evidence to support this partitioning of sources of bias when results become available.

8.7 Performance and System Audits

The following audits will be performed during this demonstration. These audits will determine if this demonstration plan is being implemented as intended.

8.7.1 Performance Audit

Performance evaluation (PE) samples will be submitted to the PCB field analytical technology for analysis. The certified concentrations of the PE samples will be used to evaluate the PCB field analytical technology. The PCB field analytical technology will analyze the PE samples periodically during the demonstration. PE samples will be obtained from Environmental Resource Associates and the U. S. EPA's Office of Solid Waste and Emergency Response's Analytical Operations and Data Quality Center.

8.7.2 On-Site System Audits

On-site system audits for field operations will be conducted as requested by the EPA project manager or technical lead. These audits will be performed by the EPA Project Manager, EPA technical lead, DOE, and/or ORNL.

8.8 Quality Assurance Reports

QA reports provide the necessary information to monitor data quality effectively. It is anticipated that the following types of QA reports will be prepared as part of this demonstration.

8.8.1 Status Reports

Through brief morning meetings on each day of the demonstration, the developer and ORNL will regularly inform the EPA and DOE project managers of the status of the project. They should discuss project progress, problems and associated corrective actions, and future scheduled activities associated with the demonstration. When problems occur, the developer and ORNL will discuss them with EPA and/or DOE, estimate the type and degree of impact, and describe the corrective actions taken to mitigate the impact and to prevent a recurrence of the problems.

8.8.2 Audit Reports

Any QA audits or inspections that take place in the field while the demonstration is being conducted will be formally reported by the auditors to EPA and DOE project managers who will forward them to the developer, Janet Wagner (CASD QA Specialist), and the ORNL program manager for appropriate actions. Informal reporting of audit results will be reported immediately to EPA and DOE.

8.9 Corrective Actions

Routine corrective action may result from common monitoring activities, such as:

- Performance evaluation audits
- Technical systems audits
- Calibration procedures

If the problem identified is technical in nature, the individual developers will be responsible for seeing that the problem is resolved. If the issue is one that is identified by ORNL, DOE, or EPA, the identifying party will be responsible for seeing that the issue is properly resolved. All corrective actions will be documented. Any occurrence that causes discrepancies from the demonstration plan will be noted in the technology verification report. The reference laboratory's SOP (See Appendix B) describes the corrective action plan for not meeting minimum QC requirements.

9.0 DATA MANAGEMENT AND ASSESSMENT

The developer, ORNL, DOE, and EPA each have distinct responsibilities for managing and analyzing demonstration data. ORNL is responsible for managing all the data and information generated during the demonstration. The developer is responsible for furnishing those records generated by the technology developer. EPA, DOE, and ORNL are responsible for analysis and verification of the data.

There are a variety of pieces of data and information that will be generated during a demonstration. Each piece of data or information identified for collection in the demonstration plan will need to be provided to ORNL.

Innovative Technology Data: The developer is responsible for obtaining, reducing, interpreting, validating, and reporting the data associated with his technology's performance. These data should be reported on the chain-of-custody. Developer results will be due to ORNL at the conclusion of a day's field activities. The developer's final report will be due to ORNL one week after the demonstration. Any discrepancies between the originally reported result and the final result must be described.

Reference Laboratory Analyses: The raw data and the validated data has already been provided to ORNL.

Other items that must be provided include:

- field notebooks;
- photographs, slides and videotapes (copies);
- results from the use of other field analytical methods;
- profiles or traces

10.0 HEALTH AND SAFETY PLAN

10.1 Introduction

This chapter describes the specific health and safety procedures that will be used during the field work at the Oak Ridge National Laboratory.

10.2 Contact Information

The ORNL project manager will be Roger Jenkins, (423) 576-8594.

The ORNL technical lead will be Amy Dindal, (423) 574-4863.

The Site Health and Safety Officer will be Fred Smith, (423) 574-4945.

The Environmental Protection Officer will be Kim Thomas, (423) 574-4947.

The ORNL Office of Safety and Health Protection Director is Ann Shirley, (423) 576-8262.

The <u>Laboratory Shift Superintendent</u> number is (423) 574-6606.

The Emergency Communications Center number is (423) 574-6646.

IN CASE OF ANY EMERGENCY, DIAL 9-1-1.

10.3 Health and Safety Plan Enforcement

ORNL project manager, field site supervisor, and site health and safety officer will be responsible for enforcing the health and safety plan. ORNL project manager will ultimately be responsible for ensuring that all demonstration participants abide by the requirements of this HASP. ORNL field site supervisor will oversee and direct field activities and is responsible for ensuring compliance with this HASP.

10.4 Site Background

The demonstration of PCB field analytical techniques will be conducted at the Oak Ridge National Laboratory (ORNL), which is managed by Lockheed Martin Energy Research Corporation, Oak Ridge, Tennessee. Oak Ridge is located a short distance from Gatlinburg and the Great Smoky Mountains National Park. Recreation areas include Big South Fork and several Tennessee Valley Authority rivers and dams. A new highway extension allows easier access to the airport, now within 20 miles of the three Oak Ridge facilities. The city of Oak Ridge is home to the American Museum of Science and Energy, the University of Tennessee Arboretum, Oak Ridge Associated Universities, and several hotels and restaurants to accommodate area visitors.

Field activities will occur at two sites at ORNL: the area west of Building 5507(outdoor site) and inside a controlled environmental atmosphere facility (chamber site) which is located in Building 5507. Building 5507 is located in a relatively secluded part of the Laboratory (see Figure 4-1). The controlled experimental atmosphere facility consists of a room-size, walk-in chamber ten feet wide and twelve feet in length with air processing equipment for temperature, humidity, and slightly subambient pressure control at air circulation flow rates up to five hundred cubic feet per minute.

10.5 Visitors

Visitors will be badged and escorted at all times by ORNL personnel. Visitors will follow standard ORNL safety and health policies and practices.

10.6 Demonstration-Specific Hazard Evaluation

The proposed demonstration activities have been evaluated by ORNL radiation protection personnel. No radiation protection hazards have been identified. PCBs issues and hazards will be controlled per ORNL procedures (Oak Ridge Reservation Polychlorinated Biphenyl Federal Facilities Compliance Agreement, ORR-PCB-FFCA). The Lockheed Martin Energy Research Corporation procedure, "EPP 3.1 Management of Polychlorinated Biphenyls" will also be followed and can be found at the following web site address: http://www-internal.ornl.gov/ORNL/directives/data/EP/WSS/eppr04a.htm.

The hazards associated with this demonstration include worker exposure to volatile organic compounds (VOCs), semivolatile organic compounds (SVOCs), and additional physical hazards associated with the technology's equipment. Plastic ground covers will be placed underneath each technology set-up, in order to collect any spills of soil or solvent. Ground covers will be replaced as necessary.

All hazardous waste generated by the technology developers will be properly disposed of by the Environmental Protection Officer. The technology developers will assist with this process by providing

accurate records of the waste contents and approximate concentrations.

10.7 Training Requirements

All technology developers must be badged and escorted by ORNL personnel at all times. The developers will be escorted in lieu of additional site-specific training.

10.8 Exposure Pathways

Exposure to VOCs and SVOCs during field activities may occur through inhalations or ingestion. The most likely exposure to VOCs and SVOCs during the demonstration will be through dermal contact. Dermal contact with contaminated soil will be prevented through the use of personal protective equipment (PPE), such as gloves. The technology developers must provide their own PPE. Although unlikely to be necessary, visitors will be provided with PPE if warranted.

10.9 Health Effects

PCBs will be the most prevalent chemical hazards at the demonstration. PCBs are:

- ► Nonflammable liquids
- ► Carcinogenic
- ► Viscous liquids with a mild, hydrocarbon odor

Some possible health effects from exposure to PCBs are: (1) irritation to the eyes and skin, possibly forming an acne condition; and (2) liver damage. If PCBs contact the skin, immediately wash the contaminated skin with soap and water. If PCBs penetrate the clothing, immediately remove the clothing and wash the skin with soap and water. Get medical attention promptly.

10.10 Physical Hazards

Physical hazards associated with field activities present a potential threat to on-site personnel. Dangers are posed by unseen obstacles, noise, heat, and poor illumination. Injuries may results from the following:

- ► Accidents due to slipping, tripping, or falling
- ► Improper lifting techniques
- ► Moving or rotating equipment
- ► Improperly maintained equipment

Injuries resulting from physical hazards can be avoided by adopting safe work practices and by using caution when working with machinery.

Fire

The following specific actions will be taken to reduce the potential for fire during site activities:

- ► No smoking within 20 feet of the site.
- ► Fire extinguishers will be maintained on-site.
- ► All personnel will be trained on the location of the portable fire extinguishers.

Mechanical, Electrical, Noise Hazards

Some technology-specific hazards may be identified once the developers set-up their equipment. Proper hazards controls (i.e., guarding or markings) or PPE (i.e., ear plugs for noise hazards) will be implemented as necessary.

Electrical cables represent a potential tripping hazards. When practical, cables will be placed in areas of low pedestrian travel. If necessary, in high pedestrian travel areas, covers will be installed over cables.

Unstable/Uneven Terrain

The terrain around Building 5507 is uneven and bumpy. Site personnel shall be aware of uneven terrain to avoid slips, trips, and falls.

Inclement Weather

The demonstration will occur the latter part of September. The possibility of inclement weather (particulary rain and thundershowers) exists. The developers should be prepared to deal with a possible inclement weather situation.

Operating temperatures in the chamber could be as low as 50° F. Developers should be prepared to work in those temperatures.

Heat Stress

Since the demonstration will occur in September, the possibility of a heat-related injury during field work is possible. Heat stress symptoms include heat cramps, heat exhaustion, and heat stroke. Heat stroke is the most serious condition and can be life-threatening. To combat heat-related injuries, ORNL will:

- ► Provide water to all demonstration participants;
- Establish a work regimen that will provide adequate rest periods;
- ▶ Provide access to air-conditioned buildings;
- ► Notify all workers of health hazards and the importance of adequate rest.

Some symptoms of heat-related injuries are pale clammy skin, sweating, headache, weakness, dizziness, and nausea. Signs of heat stroke include dry, hot, red skin, chills, and confusion. In the case of a suspected heat-related injury, try to cool the person down and contact medical help.

Insect and Other Animal Stings and Bites

A potential for insect and other animal stings or bites exists during the technology demonstration. inspect repellent may be used to minimize insect bite hazards. In the event of snake or other large animal bite, the injury should be immobilized and immediately reported to medical personnel.

10.11 Personal Protection

Personal Protective Equipment (PPE) shall be appropriate to protect against known and potential health hazards encountered during routine operation of the technology systems.

Levels of Protection

For this demonstration, Level D PPE is required. Level D provides minimal protection against chemical hazards. It consists only as a work uniform, with gloves worn, where necessary.

Protective Equipment and Clothing

Because the anticipated hazard level is low, field and chamber work will be performed using Level D protection. Level D PPE will be supplied by the individual technology developer. ORNL will provide visitors with PPE if necessary. If site conditions or the results of Industrial Hygiene monitoring indicates that additional hazards are present, PPE levels will be reconsidered.

The following is the list of protective equipment <u>required</u> for demonstration operations:

- ► Appropriate work clothes (no shorts or open-toed shoes)
- ► Disposable outer gloves.

Medical Support

A complete medical facility is located on-site in Building 4500 North. Medical help can be summoned from any laboratory phone by dialing 9-1-1. The 911 system automatically contacts the Lab Emergency Response Center and Emergency Communications Center, and Medical. Pulling a fire alarm box will summon the fire department and the laboratory shift superintendent's office.

Environmental Surveillance

The Environmental Protection Officer will be responsible for surveying the site before, during, and after the demonstration. Appropriate personnel will be on-hand to assist all demonstration participants to deal with any health or safety concerns.

10.12 Site Control

Site Control Zones

Access to the demonstration site will be unrestricted, but controlled. Any visitors to the site must be accompanied by ORNL personnel.

Safe Work Practices

Each company will provide the required training and equipment for their personnel to meet safe operating practice and procedures. The individual technology developer and their company are ultimately responsible for the safety of their workers.

The following safe work practices will be implemented at the site for worker safety:

- ► Eating, drinking, chewing tobacco, and smoking will be permitted only in designated areas;
- ► Wash facilities will be utilized by all personnel before eating, drinking, or toilet facility use;
- ▶ PPE requirements (See Section 10.11) will be followed.

Complaints

All complaints should be filed with the ORNL Field Site Supervisor (Amy Dindal). All complaints will be treated on an individual basis and be dealt with accordingly.

10.13 Radiological Hazards

The PCB-contaminated samples that will be used in this demonstration have been analyzed and found not to be radioactive. However, if an issue concerning radioactivity would occur during the demonstration ORNL-radiation procedures will be applied, where applicable.

REFERENCES

- [1] Maskarinec, M.P., et al. *Stability of Volatile Organics in Environmental Soil Samples*, ORNL/TM-12128, Oak Ridge National Laboratory, Oak Ridge, Tenn., November 1992.
- [2] U.S. Environmental Protection Agency. "Method 8081: Organochlorine Pesticides and PCBs as Aroclors by Gas Chromatography: Capillary Column Technique," in *Test Methods for Evaluating Solid Waste: Physical/Chemical Methods (SW-846)*, 3d ed., Final Update II, Office of Solid Waste and Emergency Response, Washington, D.C., September 1994.
- [3] U.S. Environmental Protection Agency. *Data Quality Objectives for Remedial Response Activities*, EPA 540/G-87/003, EPA, Washington, D.C., March 1987.
- [4] Berger, Walter, Harry McCarty, and Roy-Keith Smith, <u>Environmental Laboratory Data Evaluation</u>, Genium Publishing Corporation, Schenectady, New York, 1996.

The appendices are available in hard-copy only. Please contact Amy Dindal (423-574-4863).